Strength and Fracture Toughness of Ultrafine Grained Alumina Ceramic

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**Strength and Fracture Toughness of Ultrafine Grained Alumina Ceramic**

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Outline

- Introduction
- Experimental Procedures
- Results and Discussion
  - Bending Process
  - Fracture Morphology
  - Statistical Analysis
  - Fracture Toughness
- Conclusions
- Acknowledgements
Introduction

- **Griffith’s Theory**

\[ \sigma = \frac{K_{IC}}{Y\sqrt{a}} \]

- \( \sigma \) -- Flexure strength
- \( K_{IC} \) -- Fracture toughness
- \( Y \) -- Stress intensity factor
- \( a \) -- Critical flaw size

\[ \sigma = \sqrt{\frac{2E\gamma}{\pi a}} \quad \text{(Modified version)} \]

- \( E \) -- Elastic modulus
- \( \gamma \) -- Surface energy
- \( a \) -- Critical flaw size

Bending strength dependence on grain size of alumina

Introduction

Volume Flaw

Surface Flaw

Intergranular Fracture

Transgranular Fracture as well as Intergranular Fracture

Introduction

- Dependence of fracture toughness of alumina on grain size

Previous work on ultrafine grained alumina is limited.

Experimental Procedures

α-Al₂O₃ (TM DAR 6914)
~100nm, 99.99 %

Composition of feedstock powder

<table>
<thead>
<tr>
<th>Powder Composition</th>
<th>α - Al₂O₃ (wt. %)</th>
<th>Impurity (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>99.99</td>
<td></td>
<td>Si  Fe  Na  K  Ca  Mg</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1    8    3    2   3  1</td>
</tr>
</tbody>
</table>

Spark Plasma Sintering (SPS-825S, DR. Sinter)

As-Sintered Samples
Experimental Procedures

As-Sintered Samples

Instron universal testing machine

Dimensions of bending beam

Strain rate: 0.0015 s\(^{-1}\)

miniature 4-point bend test fixture
Experimental Procedures

- Determination of Fracture Toughness of Ultrafine Grained Alumina
  - Vickers indentation
  - Surface Crack in Flexure
  - Single-edge-V-notched beam (SEVNB)
Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness
Results and Discussion

- Average Grain Size -- 363 Å 40 nm
- Relative Density -- 99.1%
- Strength -- 434 MPa

Sample preparation methods (chamfering the edges; annealing samples for a short time before bending)
- Preloading conditions and strain rate

* Strain was calculated from gauge length (Toe compensation).
Results and Discussion

- Average Grain Size -- 320 ± 20 nm
- Relative Density -- 99.5%
- Strength -- 458MPa

* Strain was calculated from gauge length.
Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness
Results and Discussion

- Average Grain Size -- 363 40 nm
- Relative Density -- 99.1%
- Strength -- 434MPa
Results and Discussion

- Average Grain Size: $363 \pm 40$ nm
- Relative Density: 99.1%
- Strength: 434 MPa

Local magnifications:
Results and Discussion

Intergranular Fracture

Mixed Fracture (transgranular cleavage fracture + intergranular fracture)

- Average Grain Size -- 363 ± 40 nm
- Strength -- 434MPa

Intergranular Fracture

microcrack
Results and Discussion

- **Average Grain Size** -- 363 40 nm
- **Relative Density** -- 99.1%
- **Strength** -- 434MPa
Results and Discussion

- Average Grain Size -- 363 ± 40 nm
- Relative Density -- 99.1%
- Strength -- 434 MPa
Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness
Results and Discussion

Strength Distribution (Weibull Statistics)

Average Grain Size (nm)

<table>
<thead>
<tr>
<th>Grain Size</th>
<th>Number of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>300 ± 25 nm</td>
<td>11</td>
</tr>
<tr>
<td>350 ± 25 nm</td>
<td>11</td>
</tr>
<tr>
<td>400 ± 25 nm</td>
<td>13</td>
</tr>
<tr>
<td>450 ± 25 nm</td>
<td>8</td>
</tr>
<tr>
<td>600 ± 25 nm</td>
<td>10</td>
</tr>
<tr>
<td>1500 ± 100 nm</td>
<td>8</td>
</tr>
</tbody>
</table>

\[ \ln \left( \frac{1}{1 - P_n} \right) = m \ln \sigma_n - m \ln \sigma_0 \]
Results and Discussion

\[ \ln \left[ \ln \left( \frac{1}{1 - P_n} \right) \right] = m \ln \sigma_n - m \ln \sigma_0 \]

Typical Value: 5 - 20 for technical ceramics
Results and Discussion

The graph illustrates the relationship between grain size (nm) and representative strength (MPa). As the grain size decreases, the representative strength increases, reaching a peak at around 450 nm. The graph shows a clear trend indicating that smaller grain sizes lead to higher strengths.
Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness
Determination of Fracture Toughness of Ultrafine Alumina

- **Vickers indentation**
  Simple. SEM is used to measure the length

- **Surface Crack in Flexure**
  ASTM C1421-01b, 2007

- **Single-edge-V-notched beam (SEVNB)**
  ASTM STP 1409, 2002
  * It didn’t give consistent results and was abandoned in our lab.
Fracture Toughness determined by Vickers Indentation

- Surface of alumina was polished to 1µm finish before 10 indentations being made

Anstis equation

\[ K_{IC} = 0.016 \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} \]

\[ H = 1.8544 \left( \frac{P}{d^2} \right) \]

H: Hardness  E: Young’s modulus  P: Load  c: length of diagonal of indentation and radial crack


Secondary electron image

Back scattering electron image
Fracture Toughness and Hardness from Vickers Indentation

Dependence of fracture toughness and hardness of alumina on grain size
Knoop indenter precrack in a sintered alumina as photographed in optical microscope. The detection and measurement of precrack shape of width $2c$ and depth $a$ can be done in SEM or optical microscope.
Surface Crack in Flexure

\[ K_{IC} = Y\sigma\sqrt{a} \]

\( Y \): stress intensity factor, \( f(a, c, W) \)
\( \sigma \): flexure strength at fracture
\( a \): precrack depth
\( c \): half width of precrack shape
\( W \): thickness of test specimen

Flexure strength is most important to determine \( K_{IC} \)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Grain size (nm)</th>
<th>Flexure Stress (MPa)</th>
<th>Remove indenter and Damage zone?</th>
<th>Fracture Toughness (MPa·m(^{1/2})) (Surface crack in flexure)</th>
</tr>
</thead>
<tbody>
<tr>
<td>75-1</td>
<td>366 ± 28</td>
<td>243</td>
<td>Yes</td>
<td>2.61</td>
</tr>
<tr>
<td>78-1</td>
<td>622 ± 58</td>
<td>275</td>
<td>Yes</td>
<td>3.11</td>
</tr>
<tr>
<td>78-2</td>
<td>622 ± 58</td>
<td>238</td>
<td>No</td>
<td>2.78</td>
</tr>
<tr>
<td>81-1</td>
<td>870 ± 77</td>
<td>224</td>
<td>No</td>
<td>2.68</td>
</tr>
<tr>
<td>92-2</td>
<td>1402 ± 249</td>
<td>257</td>
<td>No</td>
<td>3.02</td>
</tr>
</tbody>
</table>

Typical four point bending curves
Comparison of Fracture Toughness of Alumina determined by Surface Crack in Flexure and Vickers Indentation

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Grain size (nm)</th>
<th>Fracture toughness (MPa·m$^{1/2}$) (Surface crack in flexure)</th>
<th>Fracture toughness (MPa·m$^{1/2}$) (Vickers indentation)</th>
</tr>
</thead>
<tbody>
<tr>
<td>75-1</td>
<td>366 28</td>
<td>2.61*</td>
<td>2.90 0.13</td>
</tr>
<tr>
<td>78-1</td>
<td>622 58</td>
<td>3.11*</td>
<td>2.84 0.13</td>
</tr>
<tr>
<td>78-2</td>
<td>622 58</td>
<td>2.78</td>
<td>2.84 0.13</td>
</tr>
<tr>
<td>81-1</td>
<td>870±77</td>
<td>2.68</td>
<td>2.83 0.05</td>
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<tr>
<td>92-2</td>
<td>1402 249</td>
<td>3.02</td>
<td>2.72 0.09</td>
</tr>
</tbody>
</table>

Single-Edge-V-Notched Beam (SEVNB): $K_{IC}$ varies from 3 to 5 MPa·m$^{1/2}$

Surface Crack in Flexure and Vickers indentation gave more consistent fracture toughness value in our lab.

* Removing indenter and damage zone
Conclusions

- For dense ultra-fine grained alumina, most strength values fall within the interval of 280-460 MPa. The proper selection of appropriate sample preparation methods and preloading conditions is important to achieve accurate measurements.

- No dependence of Weibull modulus on grain size can be observed.

- Cleavage, transgranular and intergranular fracture as well as microcracks, slip lines and dislocations can be observed at or near the fracture surface.

- Surface crack in flexure and Vickers indentation gives more consistent fracture toughness values than does single-edge-V-notched beam (SEVNB).
Acknowledgements

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▪ Collaborators/Contributors

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Thank You for Your Attention!